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# Micro structuring of transparent materials with NIR ns-laser pulses

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**Abstract**

A current challenge in laser processing is high precision micromachining of transparent materials, e.g. to manufacture micro-optical elements. This can be achieved amongst others by using laser induced backside wet etching. Research has been done by several groups in the last years. Most of the published results were obtained by using UV excimer lasers. Our approach deals with the implementation of the technique for NIR laser sources. We investigated the effects of different pulse widths and repetition rates on laser induced back side wet etching for 1064nm wavelength and for different absorbers.

*Keywords:* laser induced backside wet etching (LIBWE); laser ablation; surface micro-structuring; fused silica; soda lime

## 1. Motivation / State of the Art

An application with large commercial potential for modern laser surface processing is the micro-/nanostructuring of transparent materials. Especially micro-optics and -fluidics call for high precision, crack free, smooth machining techniques. Most commonly, this is done by photolithographic etching which is very well suited for mass production. Alternative methods – in particular for rapid prototyping – are direct laser writing with ultra short (ps/fs) pulsed lasers or the laser induced backside wet etching (LIBWE) technique. Using LIBWE, a variety of micro-optical structures with good optical properties have been manufactured by other groups using UV excimer lasers and mask projection [1, 2]. Few groups made an approach with Q-switched frequency-doubled or -quadrupled Nd:YAG or Nd:$YVO_4$ lasers and galvanoscanner-based point scanning systems [3, 4]. Results describing the machining of optical devices made by more affordable Q-switched NIR laser sources are very rare. Experiments using liquid gallium as absorber and NIR laser pulses in the ns-regime have been reported in the literature [5]. In our opinion, it is worthwhile to do further investigations using absorbers that are more common than gallium. This could lead to a better implementation of the technique for NIR wavelengths. An interesting and easy to handle absorber for light at 1064nm is aqueous copper (II) sulfate solution, labeled as $CuSO_4$ in the following [6].

In this paper, the possibilities of machining smooth surfaces using $CuSO_4$ and a NIR laser source are presented. Effects of different pulse widths and pulse repetition rates have been investigated. Advantages and disadvantages compared to the known UV-laser based LIBWE process are discussed. Results are also compared to those achieved

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by direct laser writing with a ps pulsed laser system and to results of experiments with other liquid absorbers for NIR laser sources.

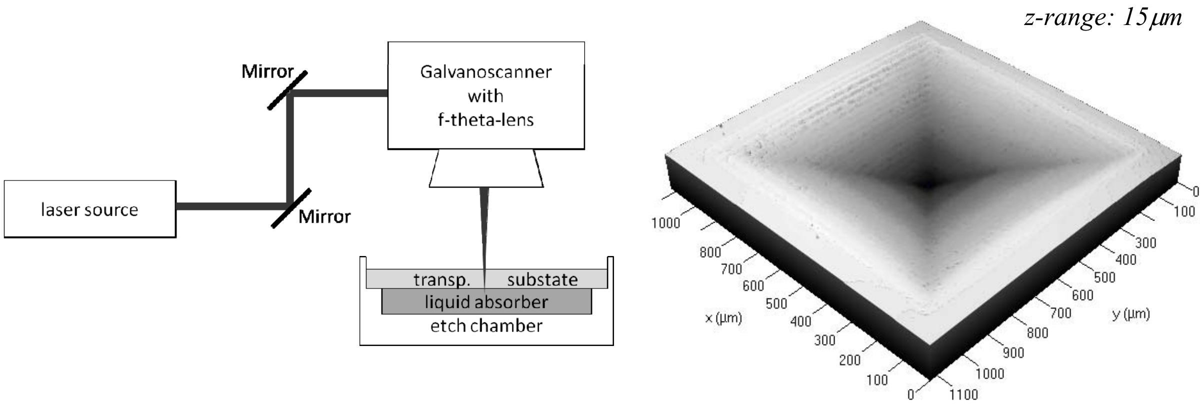


Figure 1. (a) Setup of a laser induced backside wet etching system (b) LSM image of an etched pyramidal structure in fused silica

## 2. Experimental

Figure1a shows the schematic diagram of the experimental setup. A ns-pulsed ytterbium fiber laser with 1064nm center wavelength providing 8 different pulse widths (4/8/14/20/30/50/100/200 ns FWHM) has been used.

We investigated $CuSO_4$ dissolved in de-ionized water (1.25M) [6] as liquid absorber. For comparison, a few experiments have been done by using a $CuSO_4$-based electrolyte solution (0.1M $CuSO_4$, 0.2M KNa tartrate, 0.125M NaOH and 6M HCOH) for laser induced metallic copper deposition [7] labeled as Cu-tartrate in the following and an aromatic hydrocarbon dye ADS1065A dissolved in dimethylformamide (0.002M) [8].

Both soda lime and fused silica substrates have been etched after cleaning them with ethanol. A PTFE laser etch chamber with a reservoir for the liquid absorber of about 25ml and capable of fitting 2 inch fused silica and soda lime substrates was fixed on top of a height adjustable stage. The liquid absorber was placed in contact with the downside of the transparent substrate. Laser light was irradiated from the front side of the substrate and was focused onto the solid-liquid interface, where it was absorbed by the liquid absorber. A galvanoscanner with an f-theta-lens has been used to control the scanning pattern and speed of the laser. The topography of the etched features was quantitatively measured with a confocal laser scanning microscope (LSM). An example of a LIBWE structure measured with an LSM is shown in Figure1b. To perform in addition also direct laser ablation, a picoseconds MOPA laser system with 532nm wavelength irradiation and a pulse width of 10ps (FWHM) has been used in combination with a similar galvanoscanner.

## 3. Results and Discussion

Well-defined etching grooves without micro cracks in the vicinity of the groove with app. 30µm width and app. 12µm depth were achieved in both fused silica and soda lime glass by NIR laser irradiation and by using $CuSO_4$ (see Figure2a). The cross section of the grooves is V-shaped due to the Gaussian intensity profile of the laser beam.

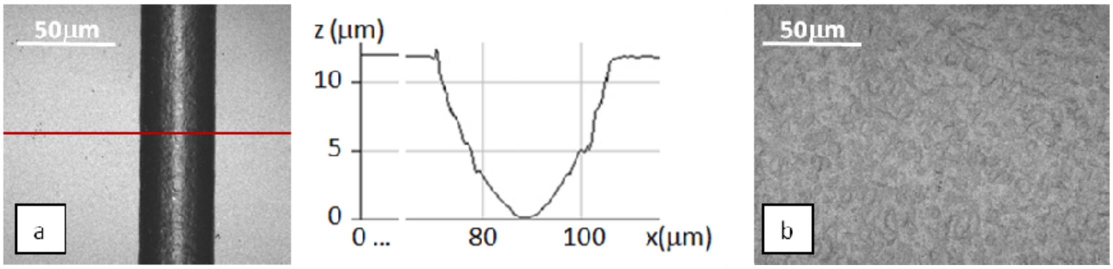


Figure 2. (a) Groove and corresponding cross section (position indicated by the horizontal line in the right image) etched in soda lime glass with 5.5J/cm$^2$ fluence, 100kHz pulse repetition rate, 20ns pulse width (FWHM), 40mm/s scan speed, 250 iterations (b) Etched grid pattern in fused silica with measured $R_a$-value of ca. 130nm.

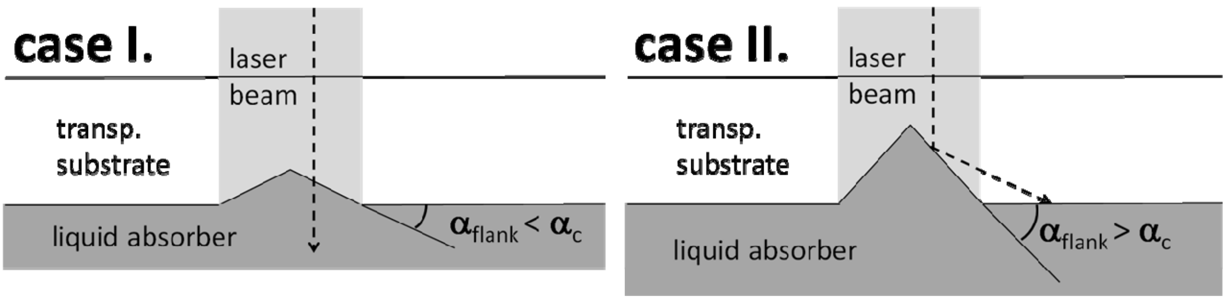


Figure 3. Case I: flank angle of groove is smaller than the critical angle of total reflection; irradiated light reaches the absorber directly. Case II: flank angle of groove is larger than the critical angle of total reflection; irradiated light is total reflected and causes chipping effects at groove borders.

0.5x0.5mm$^2$ squares with depths of app. 10μm have been fabricated by scanning a grid pattern based on overlapping single lines. LSM-measurements have been performed by analyzing an 180x180μm$^2$ area in order to compare the surface roughness of different laser and scanning parameters. Depending on scan velocity, pulse repetition rate, laser power and scan pattern arrangement, smooth surfaces with $R_a$-values down to 130nm (see Figure2b) have been achieved as well as honeycomb-like structures with $R_a$-values up to 2μm (see Figure7a).

For comparison: similar experiments done by direct laser ablation using a ps-pulsed laser system lead to $R_a$-values of about 300nm for sapphire and of about 400nm for soda lime. The smoothness achieved by LIBWE could not be attained.

The aspect ratio of LIBWE structures as fabricated with our experimental setup, however, is limited by total reflection between substrate and liquid absorber. If the flank angle $\alpha_{flank}$ of etched grooves is larger than the critical angle for total reflection $\alpha_c$, the laser beam is totally reflected at the interface and cannot reach the absorber (see Figure3). The critical angle for total reflection $\alpha_c$ is calculated as app. 61° for soda lime and app. 65° for fused silica, if aqueous $CuSO_4$ is treated as pure water. Grooves with a larger flank angle show chipping effects at the groove borders due to these total reflection effects (see Figure4b and d). Despite this limitation, various structural designs are possible, for example pyramidal structures (see Figure1b). Total reflection could possibly be diminished by performing an index match between substrate and liquid absorber. However, it is probably difficult to find a suitable substance which features a corresponding refractive index and does not inhibit the solubility of $CuSO_4$ or disturb the chemical and physical ablation processes.

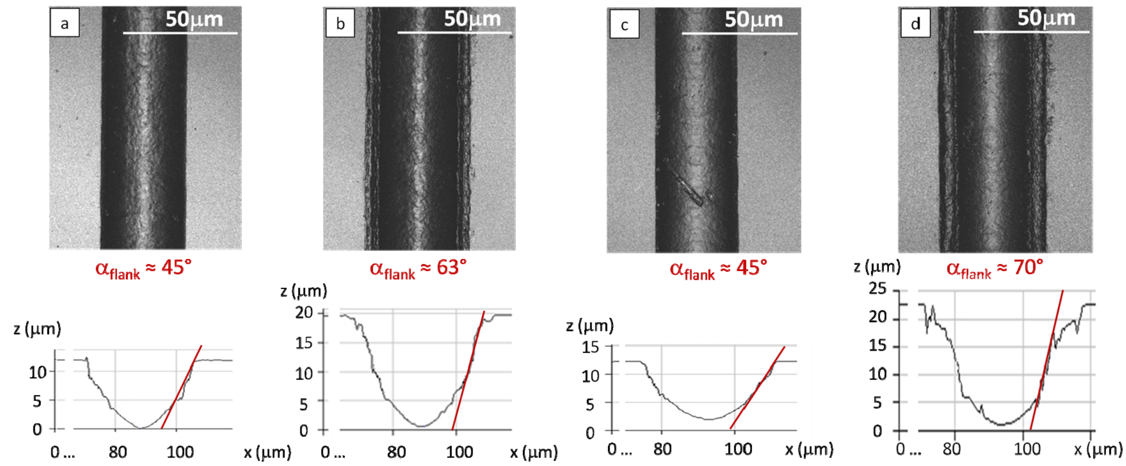


Figure 4. (a) Groove and corresponding cross section with estimated flank angle etched in soda lime glass with 5.5J/cm$^2$ fluence, 100kHz pulse repetition rate, 20ns pulse width (FWHM), 40mm/s scan speed, 250 iterations and 500 iterations (b) respectively. (c) Groove etched in fused silica with 4.8J/cm$^2$ fluence, 100kHz pulse repetition rate, 20ns pulse width (FWHM) 40mm/s scan speed, 500 iterations and 1000 iterations (d) respectively. Chipping effects at the groove borders due to total reflection effects can be observed in (b) and (d).

The results shown previously (see Figs. 2 and 4) show that a smooth ablation is possible with 20ns pulse width and a pulse repetition rate of 100 kHz. In the following we want to discuss the influence of the repetition rate and the pulse width on the ablation quality. In order to compare different pulse repetition rates, grooves have been etched with different repetition rates but identical fluence values (4 J/cm$^2$) by adjusting the mean power. The scan speed has been adapted according to the chosen repetition rate, such that the overlap of the irradiated surface area per pulse was the same for every groove. The grooves etched with rather low pulse repetition rates (50 to 150 kHz) revealed homogenous ablation rates and a maximum in ablation efficiency at around 100 kHz. The higher the pulse repetition rates, the more the ablation rate decreases again and melting effects can be observed (see Figs. 5 and 6).

In addition, grooves have been etched with pulse widths of 4, 8, 14, 20, 30, 50, 100 and 200 ns (FWHM) by choosing fluence values and scanning parameters as described above. We observed a similar tendency of the ablation rate in function of the pulse width as for the ablation rate in function of the pulse repetition rate. The ablation efficiency reaches a maximum with pulse widths at about 20 to 50 ns. With 4-ns-pulses, ablation was not possible, while pulse widths above 100 ns lead to melting effects at the surface instead of a smooth ablation.

The detailed processes leading to these effects for varying pulse widths and repetition rates are not clear yet. Formation of gas bubbles in the liquid absorber for example can be observed with increasing pulse width and/or pulse repetition rate. Such gas bubbles may affect the ablation mechanism. It is possible that a recirculation of the liquid absorber and the resulting dissipation of bubbles away from the process area would lead to better results for longer pulse widths or higher pulse repetition rates. In general, the possibility to be able to use higher pulse repetition rates and therefore higher scan speeds with similar ablation quality is interesting regarding the overall processing time.

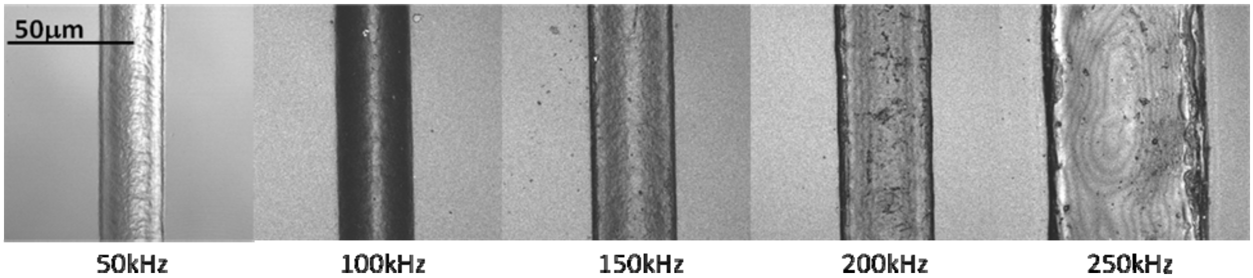


Figure 5. Grooves etched in soda lime glass in order to compare the effect of the pulse repetition rate. Parameters for every groove: 4J/cm$^2$ fluence, 20ns pulse width (FWHM), 40mm/s scan speed and 200 iterations. Scan speed from left to right: 20mm/s, 40mm/s, 60mm/s, 80mm/s, 100mm/s. Visible melting effects occur at 250 kHz pulse repetition rate.

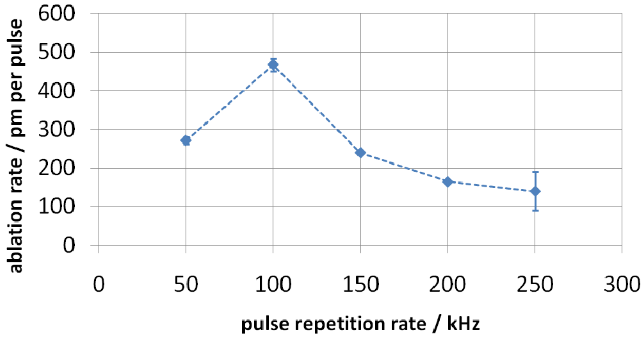


Figure 6. Ablation rates in function of the pulse repetition rate. The dashed line serves as guide for the eye. A maximum in ablation efficiency is observed at around 100 kHz. Visible melting effects occur at 250 kHz which lead to heterogeneity and therefore a higher standard deviation of the measured ablation rates. For 50, 150, and 200 kHz repetition rate the error bars are too small to be visible.

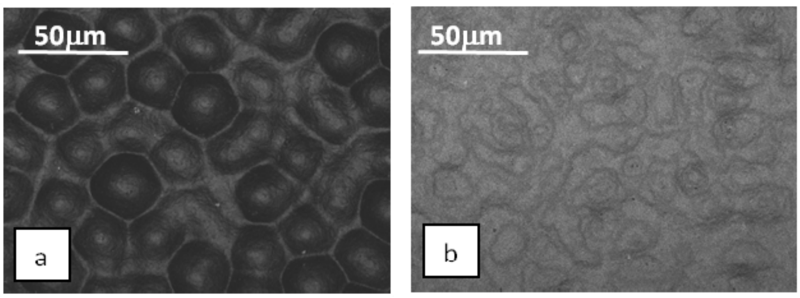


Figure 7. (a) Etched grid pattern in fused silica with 4J/cm$^2$ fluence, 100kHz pulse repetition rate, 20ns pulse width, 20mm/s scan speed with measured $R_a$-value of ca. 2µm and (b) 80mm/s scan speed with measured $R_a$-value of ca. 290nm respectively. The geometry of the scanned grid pattern is the same for both structures.

Regarding the surface smoothness, not only laser parameters like pulse width and repetition rate play an important role for the result, but also the scanning strategy. Based on the same laser parameters and therefore the same ablation effect, a variety of topographic structures can be achieved by scanning grid patterns with different scan velocities, mesh widths and grid angles (see Figure7 for two examples). Scan designs however are limited by the resolution of the scanning soft- and hardware, in particular concerning the mesh width of the grid patterns. As the surface roughness is found to be minimal with the smallest mesh width, it is useful to adjust the spot size with appropriate scan optics, if required.

Finally we briefly want to discuss the ablation using $CuSO_4$ compared to other absorbers for NIR light. Ablation by using Cu-tartrate as liquid absorber was possible with a much higher efficiency compared to $CuSO_4$, but only with much higher surface roughness. As described elsewhere [9], the ablation process for near infrared laser irradiation for $CuSO_4$ and Cu-tartrate is driven by different copper species at the glass surface that absorb the laser pulses. The higher efficiency in conjunction with the lower surface quality in the case of Cu-tartrate is attributed to the fact, that metallic copper clusters absorb the light, whereas in the case of $CuSO_4$, the light is absorbed by isolated $Cu^{2+}$-ions [9]. The assumption that absorbed species at the surface are responsible for ablation is corroborated by the experiments using ADS1065A. Even though this dye is a strong absorber for light at 1064nm wavelength, only melting or cracking and subsequent damage of the substrates, but no controlled ablation was observed.

## 4. Summary and Outlook

Our results indicate that $CuSO_4$ seems to be a well suited alternative to liquid metal absorbers for LIBWE using NIR ns laser pulses. Best results were obtained with pulse widths of 20 ns to 50 ns and pulse repetition rates of 100 kHz. By choosing an appropriate scanning strategy, surfaces with different surface topographies and roughness values can be achieved. However, the surface smoothness achieved up to now has still to be improved to be competitive with LIBWE using UV-excimer lasers. This should be achievable by further improving the process parameters. In addition, the influence of the pulse width on the surface roughness has to be further examined.

The huge advantage of our approach using NIR laser pulses compared to LIBWE using UV-excimer lasers is the fact that it is possible to machine substrates which are not transparent for UV radiation, for example soda lime glass. Other advantages of our setup along with the economic factor are the direct scan method which doesn't need projection masks, and the easy to handle liquid absorber $CuSO_4$.

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